

# **Nano-Sized Grain Refinement Using Friction Stir Processing**

**by Brian Thompson, Kevin Doherty, Jianqing Su, and Rajiv Mishra**

**ARL-RP-422**

**March 2013**

*A reprint from Proceedings of TMS 142nd Annual Meeting and Exhibition, San Antonio, TX, 3–7 March 2013.*

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REPORT DOCUMENTATION PAGE				Form Approved OMB No. 0704-0188	
Public reporting burden for this collection of information is estimated to average 1 hour per response, including the time for reviewing instructions, searching existing data sources, gathering and maintaining the data needed, and completing and reviewing the collection information. Send comments regarding this burden estimate or any other aspect of this collection of information, including suggestions for reducing the burden, to Department of Defense, Washington Headquarters Services, Directorate for Information Operations and Reports (0704-0188), 1215 Jefferson Davis Highway, Suite 1204, Arlington, VA 22202-4302. Respondents should be aware that notwithstanding any other provision of law, no person shall be subject to any penalty for failing to comply with a collection of information if it does not display a currently valid OMB control number. <b>PLEASE DO NOT RETURN YOUR FORM TO THE ABOVE ADDRESS.</b>					
1. REPORT DATE (DD-MM-YYYY) March 2013		2. REPORT TYPE Reprint		3. DATES COVERED (From - To) June 2009–November 2012	
4. TITLE AND SUBTITLE Nano-Sized Grain Refinement Using Friction Stir Processing				5a. CONTRACT NUMBER DAAD19-03-2-0002	
				5b. GRANT NUMBER	
				5c. PROGRAM ELEMENT NUMBER	
6. AUTHOR(S) Brian Thompson,* Kevin Doherty, Jianqing Su, <sup>†</sup> and Rajiv Mishra <sup>†</sup>				5d. PROJECT NUMBER	
				5e. TASK NUMBER	
				5f. WORK UNIT NUMBER	
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) U.S. Army Research Laboratory ATTN: RDRL-WMM-F Aberdeen Proving Ground, MD 21005-5069				8. PERFORMING ORGANIZATION REPORT NUMBER ARL-RP-422	
9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)				10. SPONSOR/MONITOR'S ACRONYM(S)	
				11. SPONSOR/MONITOR'S REPORT NUMBER(S)	
12. DISTRIBUTION/AVAILABILITY STATEMENT Approved for public release; distribution is unlimited.					
13. SUPPLEMENTARY NOTES A reprint from <i>Proceedings of TMS 142nd Annual Meeting and Exhibition</i> , San Antonio, TX, 3–7 March 2013. *EWI, 1250 Arthur E. Adams Dr., Columbus, OH 43221 <sup>†</sup> University of North Texas, 1155 Union Circle, No. 305310, Denton, TX 76203					
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15. SUBJECT TERMS magnesium, friction stir processing, nano-sized grains, active cooling, dynamic recrystallization					
16. SECURITY CLASSIFICATION OF:			17. LIMITATION OF ABSTRACT  UU	18. NUMBER OF PAGES  16	19a. NAME OF RESPONSIBLE PERSON Kevin Doherty
a. REPORT Unclassified	b. ABSTRACT Unclassified	c. THIS PAGE Unclassified			19b. TELEPHONE NUMBER (Include area code) 410-306-0871

## **NANO-SIZED GRAIN REFINEMENT USING FRICTION STIR PROCESSING**

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Keywords: Friction Stir Processing, Magnesium, Nano-size grains

### **Abstract**

A key characteristic of a friction stir weld is a very fine grain microstructure produced as a result of dynamic recrystallization. The friction stir processing (FSP) technique was applied to modify the through thickness microstructure of a monolithic plate of a magnesium alloy. Grain structure refinement in these alloys could have a significant impact on their strength and ductility opening up their use for high performance defense applications. EWI has been investigating the use of the FSP technique to achieve nano-sized grains in a magnesium alloy, AZ31B. Heat input estimations have enabled the prediction of welding parameters and tool geometries that could achieve significant grain refinement. This presentation will summarize the experimental procedures using active cooling and theoretical efforts undertaken in order to achieve an average stir zone grain size of 500 nm. This work was performed under a cooperative agreement between EWI and the U.S. Army Research Laboratory.

### **Introduction**

Magnesium (Mg) alloys have been widely used for structural components in the automotive, aerospace and electronics industry due to their low density, high strength to stiffness ratio, good damping capacity, diecastability and recycling. However Mg alloys also exhibit low formability because of their hexagonal close-packed crystal (HCP) structure with only two independent operative basal slip systems at room temperature. It is well accepted that grain refinement may increase strength and ductility of Mg alloys, potentially leading to wider use in a variety of industries including the defense industry.

In order to obtain a microstructure with improved mechanical behavior, several methods based on plastic deformation have been developed for grain refinement in Mg alloys such as rolling, Equal Channel Angular Processing (ECAP) and Equal Channel Angular Extrusion (ECAE). Recently, friction stir processing (FSP) has emerged as an effective tool for grain refinement [1]. FSP is a solid-state process developed on the basis of the friction stir welding (FSW) technique invented by The Welding Institute (TWI) in 1991 [2]. During FSP, heat is generated by the friction between the tool and the work piece and by the plastic deformation occurring around the tool. The material within the processed zone undergoes intense plastic deformation resulting in a dynamically recrystallized fine grain structure. Grain refinement by FSP in Mg alloys has been reported by several researchers [3-9]. It has been demonstrated that the processed grain

structures strongly depended on the processing conditions including processing parameters, tool geometry and cooling rate.

In this work, a systematic investigation was performed to study the effects of processing parameters on the resulting microstructure in AZ31B Mg alloy. Ranges of processing conditions were tested using two different FSP tools in ambient conditions and cooled conditions using a liquid nitrogen cooled anvil. The tool designs and processing conditions selected were aimed at reducing the grain size of the AZ31B Mg alloy to create an Ultra-Fine Grain (UFG) structure averaging 500-nm. Using optical microscopy and the electron back-scatter diffraction (EBSD) technique, grain size, grain structure, grain boundary characteristics and micro-texture were examined for the processed samples. On the basis of measured grain size, combined with the *Zener-Holloman* parameter, the peak temperatures in the nugget zone were calculated and compared with experimentally measured temperatures. The relationships between processing parameters, tool geometry, grain size, and temperature were also established in this work.

## **Experimental Procedure**

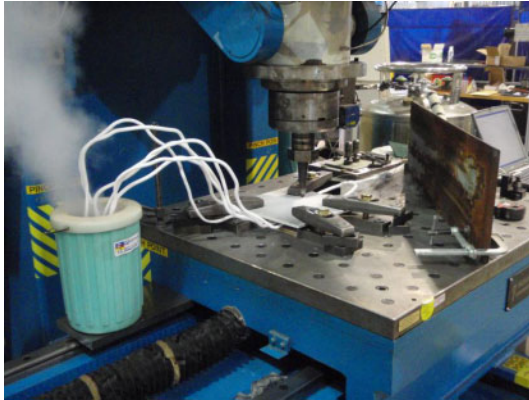
### **Friction Stir Processing Trials**

The friction stir processing trials were conducted at EWI on a NOVA-TECH Engineering Friction Stir Welding machine furnished by the U.S. Government. Two tools were designed and fabricated from 350M steel and heat treated to an approximate hardness of 60 HRC. Tool 52283-T02 incorporated a left hand, stepped spiral thread on a truncated cone type pin with negative profile scroll features on the shoulder. Tool 52283-T03 also included negative profile scroll features on the shoulder, but incorporated three blades in place of a single pin. Both tools were designed to process down to a thickness of 2.3-mm. Tool 52283-T03 was the larger of the two tools with a shoulder diameter of 17.8-mm, while tool 52283-T02 had a shoulder diameter of 11.1-mm. The aggressive features on each tool were designed in an effort to promote as much grain refinement as possible.

In order to manage the cooling rate of the AZ31B Mg alloy during processing, a copper anvil capable of being cooled by liquid nitrogen was designed and fabricated. Two inlets allowed liquid nitrogen to flow into four channels underneath the top surface of a copper block. Six outlets directly opposite the two inlets allowed the nitrogen to flow out of the copper block and be captured in an insulated bucket. A single groove machined into the surface of the copper block accommodated a single Type K thermocouple. This thermocouple was used to monitor the temperature at the root of each friction stir processing trial. A small hole was drilled into the bottom of each plate to a depth of approximately 4-mm, placing the thermocouple tip just underneath the processing trial. Surface cooling was added through the use of two copper bars lying adjacent and parallel to the processing direction. These copper bars contained a hole through which liquid nitrogen flowed and then emptied into an insulated bucket. An image of the experimental setup without the top surface copper bars is displayed in Figure 1.

The 6.3-mm thick wrought AZ31B Mg alloy plate used in this work was cut into 305-mm square plates for welding. All cooled processing trials were conducted in a single linear pass positioned on the center of the plate. Temperature data was recorded four times a second using an Omega

eight channel USB thermocouple data acquisition module. Prior to conducting processing trials, test cooling runs were conducted to establish a base line temperature to which the anvil could be reduced (-180°C). This temperature was then used as the target temperature at which to cool the anvil prior to and after each processing pass.



**Figure 1. FSP Experimental Setup**

The processing parameters for each tool and condition are summarized in Table I. After establishing initial parameters through scaling trails, the travel speed was varied while the RPM remained constant for each tool. This resulted in a selection of seven welding conditions between both tools. The variation in travel speeds was designed to provide a selection of heat inputs ranging from low to high. Each of the processing parameters for each tool were run in an ambient condition (no liquid nitrogen cooling) and a cooled condition (with liquid nitrogen cooling) on the same copper anvil.

**Table I. Selected Processing Parameters for Each Tool Design**

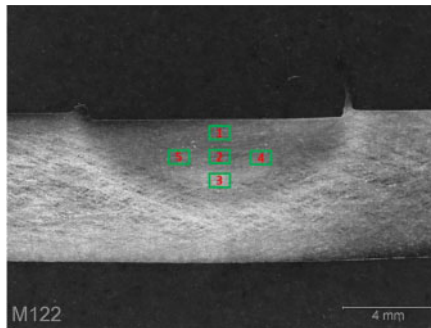
Tool No.	Spindle Speed (RPM)	Travel Speed (mm/min)	Heat Input	Ambient Cross-Section No.	Chilled Cross-Section No.
52283-T02	1200	762	Low	M16	M123
	1200	508	Medium	M15	M121
	1200	356	High	M14	M122
52283-T03	1000	762	Low	M12	M128
	1000	635	Moderate	M13	M127
	1000	508	Medium	M11	M129
	1000	381	High	M17	M130

#### Microstructural Analysis

Post-processing, each weld was cross-sectioned once for microstructural analysis resulting in 14 samples (7 ambient and 7 cooled). Each cross-section was labeled with a unique identifier as listed in Table I. The samples were polished with SiC papers using alcohol as a lubricant and

were swab etched with Kroll's solution. Optical microscopy was conducted at EWI on all samples using an Olympus BX51 microscope. Average grain size was measured over a sampling of 10 grains located in the center of the nugget zone following ASTM E1382 (Region 2, Figure 2). After completion of the optical microscopy, all seven cross-sections from the cooled processing trials were sent to the University of North Texas (UNT) for EBSD analysis.

In preparation for EBSD analysis, the seven samples were re-polished once received at UNT. The cross-sections were ground on progressively finer SiC papers to a 2400 grit finish and then polished with 0.1  $\mu\text{m}$  alumina solution. To obtain high quality Kikuchi diffraction patterns, electro polishing was carried out with a solution of 40 pct phosphoric acid and 60 pct ethanol at  $\sim 0^\circ\text{C}$  and using a voltage of 2 to 3 V. EBSD analysis was conducted at UNT with a FEI Nova NanoSEM 230 equipped with the TSL OIM<sup>TM</sup> EBSD system. A scanning step size of 0.5  $\mu\text{m}$  was used for all the scans. The error in the determination of the crystallographic orientation of each grain was less than  $2^\circ$ . In order to obtain a highly reliable picture of the microstructure, the resulting data was subjected to the following clean-up procedure: (1) grain dilatation with a grain tolerance angle of  $5^\circ$ , and (2) each grain contains at least two scan points. The different locations in the nugget zone examined using OIM are illustrated in Figure 2.



**Figure 2. Optical Microscopy and OIM Scan Locations in the Nugget Zone. 1-Top, 2-Center, 3-Bottom, 4-Advancing Side and 5-Retreating Side.**

## **Results and Discussion**

### **Friction Stir Processing Trials**

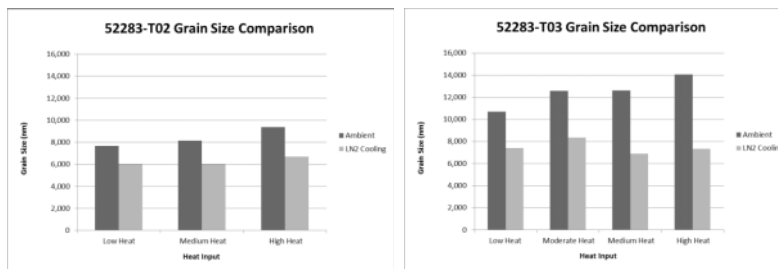
During the processing trials, temperature measurements were only recorded on the trials cooled by liquid nitrogen. Due to the proximity of the thermocouple tip to the processed zone, damage to the thermocouples resulted during two processing trials: M123 and M130. For tool 52283-T02, the peak temperature data was inconclusive for the low heat input trial (M123), reached  $\sim 200^\circ\text{C}$  for the medium heat input trial (M121), and reached  $\sim 225^\circ\text{C}$  for the high heat input trial (M122). Cooling back down to  $0^\circ\text{C}$  from the peak temperature reached during each of these trials took approximately four seconds. For tool 52283-T03 the peak temperature reached  $\sim 250^\circ\text{C}$  for the low heat input trial (M128), reached  $\sim 270^\circ\text{C}$  for the moderate heat input trial (M127), reached  $\sim 300^\circ\text{C}$  for the medium heat input trials (M129), and was inconclusive for the



high heat input trial due to the destruction of the thermocouple. The low heat input and moderate heat input trials took approximately four seconds to cool back down to 0°C from peak temperature while the medium heat input trial took approximately seven seconds.

### Optical Microscopy

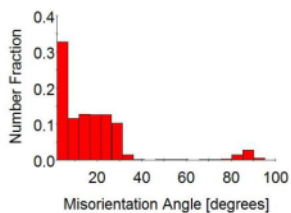
Optical analysis of the weld cross-sections revealed a reduction in grain size when comparing ambient processing trials to those performed with liquid nitrogen cooling. Ambient trials with tool 52283-T02 resulted in an increasing average grain size when increasing the heat input of the welding conditions. The low heat input trial averaged a grain size of approximately 7,600-nm while the high heat input trial averaged a grain size of approximately 9,300-nm (Figure 3). When these same welding conditions were cooled using liquid nitrogen, the average grain size dropped to approximately 6,300-nm. A similar trend was observed in the welding conditions selected for tool 52283-T03. Average grain size for the ambient trails ranged from 10,700-nm to 14,000-nm, for the low to high heat inputs respectively (Figure 3). When liquid nitrogen cooling was applied to these same welding conditions, the average measured grain size dropped to approximately 7,500-nm.



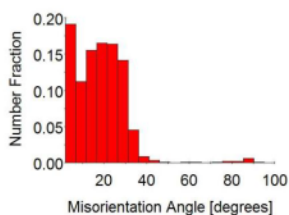
**Figure 3. Grain Size Comparisons for each Tool Based Upon Optical Measurements**

### Orientation Imaging Microscopy

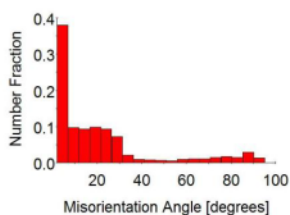
As measured by the TSL OIM™ EBSD system, the average grain sizes in samples M122, M121 and M123 processed by tool 52283-T02 are 2,900-nm, 2,400-nm, and 2,200-nm. Larger grains of 3,200-nm, 3,200-nm, 3,200-nm and 3,300-nm in size were obtained in samples M130, M129, M127 and M128 processed by tool 52283-T03. The present results show that with an increase in tool traverse speed, the processed grain size decreases in the samples processed by tool 52283-T02. However, tool traverse speed has no effect on grain size in the samples processed by tool 52283-T03. The disparity in the grain size measurements between those conducted optically following ASTM E1382 and those conducted using OIM, can be attributed to the inherent resolution and accuracy of the TSL OIM™ EBSD system. As a result, the optical grain size measurements have been used as reference only in preference of those grain sizes observed through OIM.



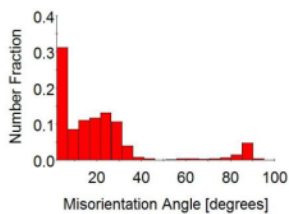
a)



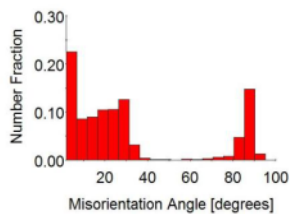
b)



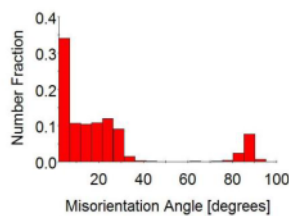
c)



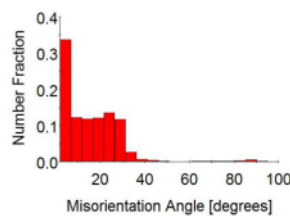
d)



d)



f)



g)

**Figure 4. Grain Boundary Misorientation Distributions in Samples a) M122, b) M121, c) M123, d) M130, e) M129, f) M127 and g) M128**

In all the cases, the liquid nitrogen cooled samples show similar grain boundary character (Figure 4). The grain boundary misorientation distributions range mainly from 2-35°, followed by a wide gap in the interval of ~35-80°, and then show a peak in ~80-90° which may correspond to the formation of twins with misorientation close to the ideal 86° <11-20> relationship. All of the processed samples have a strong fiber texture with the c-axes of the grains about 35-55° away from processing direction.

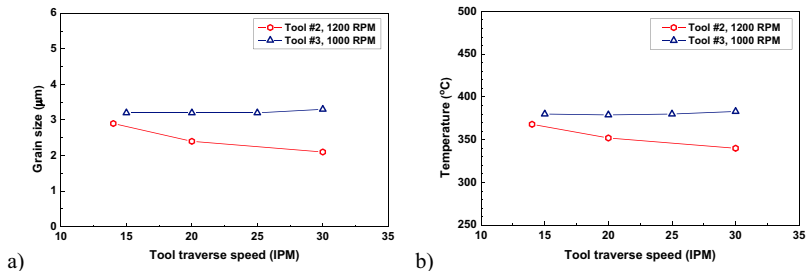
**Temperature Calculation** The Zener–Holloman parameter, combining working temperature and strain rate, has been shown to impose influence on the resulting grain size in extruded Mg based alloys [10]. This parameter appears to be useful in predicting the resulting grain size. Similarly, using the measured grain size, working temperature can be calculated using the following equation.

$$T = \frac{Q}{R(\ln Z - \ln \dot{\epsilon})} \quad (1)$$

The measured grain size and calculated peak temperature in the nugget for the samples processed by different tools under various processing parameters are summarized in Table II. The influence of the processing parameters on resulting grain size and temperature is displayed in Figure 5.

**Table II. Summary of Grain Size and Peak Temperature at Nugget Center**

Tool No.	Chilled Cross-Section No.	Spindle Speed (RPM)	Travel Speed (mm/min)	Grain Size (nm)	Calculated Peak Temperature (°C)	Measured Root Temperature (°C)
52283-T02	M123	1200	762	2,100	340	N/A
	M121	1200	508	2,400	352	200
	M122	1200	356	2,900	368	225
52283-T03	M128	1000	762	3,300	383	250
	M127	1000	635	3,200	380	270
	M129	1000	508	3,200	379	300
	M130	1000	381	3,200	380	N/A



**Figure 5. Effect of Tool Traverse Speed (IPM) on a) grain size and b) temperature**

For tool 52283-T02, an increase in the tool traverse speed causes a decrease in the peak temperature resulting in a finer grain structure in the nugget zone. This result is consistent with

those reported by many researchers in different metals and alloys. However, it is noted that there is almost no difference in grain size in the samples processed by tool 52283-T03 under different tool traverse speeds. This result suggests that to discuss the effect of processing parameters on the resulting microstructure, the tool geometry must be considered. It is also believed that a lower tool rotation rate with tool 52283-T02 would produce a finer grain structure, especially using liquid nitrogen cooling.

Tool Geometry Effects on Heat Input During FSP During FSW or FSP, peak temperature in the nugget zone plays a vital role in determining the processed grain structure. The most important factor in controlling the peak temperature is heat input. Considering power consumed by the machine during the welding process, Khandkar et al. [12] measured the torque and the rotation rate during welding, from which the radial heat flux  $\dot{q}(r)$  was calculated as:

$$\dot{q}(r) = \frac{P_{av} r}{\frac{2}{3} \pi R_{Shoulder}^3 + 2 \pi R_{Pin}^2 H_{Pin}} \quad (2)$$

where  $P_{av}$  is the average power input calculated from the measured torque and rotation rate,  $R_{Shoulder}$  is the shoulder radius,  $R_{Pin}$  is the pin radius and  $H_{Pin}$  is the pin height. Thus, heat generation is closely related to tool geometry. As demonstrated through experimental trials, the larger average grain sizes for the 52283-T03 tool (7,500-nm) are a result of its larger geometry and therefore higher heat input even though the RPM was less than that of the 52283-T02 tool (6,300-nm). Therefore, it is believed that a tool with a smaller shoulder and pin can decrease heat input caused by friction and plastic deformation. This combined with low heat input generating parameters, could result in finer grain structures created during FSP, especially when using liquid nitrogen cooling.

Fabrication of UFG Structures The purpose of this work was to produce UFG structures averaging 500-nm in size in AZ31B Mg alloy using FSP. On the bases of the relationships between grain size ( $d$ )-the Zener-Hollomon parameter ( $Z$ ) and the Zener-Hollomon parameter ( $Z$ )-peak temperature ( $T$ ) in the FSP AZ31B Mg alloy [11], the Zener-Hollomon parameter ( $Z$ ) and peak temperature ( $T$ ) can be calculated for a grain structure averaging 500-nm in size. For tool 52283-T02, which has demonstrated a higher potential to produce a finer grain structure due to its smaller geometry, the calculated plots for the relationships between  $d$ - $Z$  and  $d$ - $T$  are displayed in Figure 6.

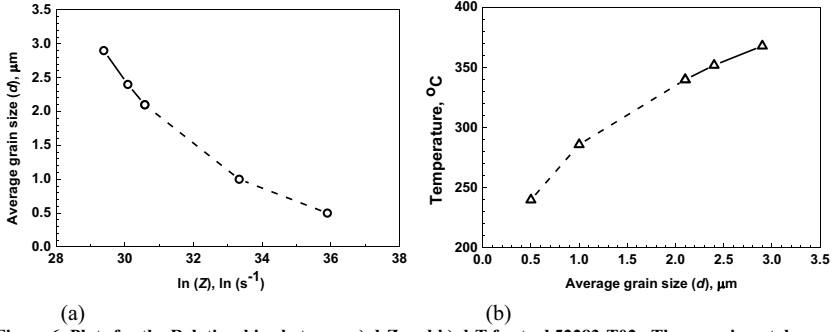


Figure 6. Plots for the Relationships between a)  $d$ - $Z$  and b)  $d$ - $T$  for tool 52283-T02. The experimental range already explored is depicted by continuous lines and the dash line represents extrapolation using the constitutive relationships

### Future Work

To create an UFG structure averaging 500-nm in size, a lower peak temperature of about  $240^{\circ}\text{C}$  is required. In the present results, average grain size as small as 2,100-nm was obtained by using tool 52283-T02, which corresponds to a peak temperature of  $\sim 340^{\circ}\text{C}$  in the nugget zone. Decreasing the peak temperature during FSP could be accomplished by increasing the cooling rate and/or decreasing the heat input. Since liquid nitrogen cooling has been used to control the cooling rate in this present work, it would be very difficult to further increase the cooling rate while using the tools as currently designed. Therefore, to obtain a finer grain structure, a reduction in heat input must be achieved through tool design and processing parameter modification. From the relationships of  $d$ - $Z$  and  $Z$ - $T$  [11], the temperature can be calculated as;

$$T = \frac{Q}{R \left[ \frac{9.0 - \ln d}{0.27} - \ln(2\pi R_m \frac{r_s}{L_s}) \right]} \quad (3)$$

For a small tool with a fast cooling rate, the  $r_s/L_s$  can be regarded as the ratio of tool pin radius to tool pin length. Using the current dimensions of tool 52283-T02, a 500-nm grain size corresponds to a temperature of  $\sim 240^{\circ}\text{C}$ . In the current trials, this tool was able to produce a 2,100-nm average grain size at a peak temperature of  $\sim 340^{\circ}\text{C}$ . This means that in order to decrease the peak temperature from 340 to  $240^{\circ}\text{C}$  using the current processing conditions, the dimensions of tool 52283-T02 would need to be decreased by 1/2 to 2/3. However, this could have practical implications in terms of volume of processed material per unit production time.

### Conclusions

- 1) Liquid nitrogen cooling of FSP trials in AZ31B Mg alloy resulted in lower average grain sizes as compared to those produced in ambient temperatures.
- 2) OIM resulted in a more accurate measurement of average grain size than optical methods.

- 3) Due to proximity to the weld, the measured temperatures in the root of the nugget zone were less than those calculated; however they both exhibit an upward trend with increasing heat input and tool geometry.
- 4) Fine grain structures of 2,900, 2,400 and 2,100-nm were observed in specimens processed by tool 52283-T02.
- 5) In comparison to tool 52283-T02, tool 52283-T03 resulted in larger average grain sizes of 3,300, 3,200, 3,200 and 3,200-nm.
- 6) Most of the grain structures consisted of high-angle boundaries but have distinct textural components. The grain boundary misorientation distributions range mainly from 2-35°, followed by a wide gap in the interval of ~35-80°, then show a peak in ~80-90° which may correspond to formation of twins with misorientation close to the ideal 86° <11-20> relationship.
- 7) The FSP samples showed strong fiber textures with the c-axes of the grains about 35°-55° away from the processing direction.
- 8) The calculated temperatures ranged from 340 to 368°C for the samples processed by tool 52283-T02, and are about 380°C for the samples processed by tool 52283-T03.
- 9) With an increase in tool traverse speed, grain size decreases in the nugget zone in samples processed by tool 52283-T02. However, there is almost no change in grain size in the samples processed by tool 52283-T03, suggesting a complex effect of strain and temperature caused by tool geometry.
- 10) To produce UFG structures averaging 500-nm in size, smaller tools with dimensions 2/3~1/2 those of tool 52283-T02, as well as a lower tool rotation rate are suggested.

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